PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference 3154PTWO/er	**FOR FURTHER ACTION**	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
International application No. PCT/EP 03/00546	International filing date *(day/month/year)* 21.01.2003	Priority date *(day/month/year)* 21.01.2002
International Patent Classification (IPC) or both national classification and IPC G01N23/20		
Applicant BERTI, Giovanni		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 7 sheets, including this cover sheet.

   ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consist of a total of 5 sheets.

3. This report contains indications relating to the following items:

   - I ☒ Basis of the opinion
   - II ☐ Priority
   - III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   - IV ☐ Lack of unity of invention
   - V ☒ Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
   - VI ☐ Certain documents cited
   - VII ☐ Certain defects in the international application
   - VIII ☐ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
20.08.2003	29.03.2004
Name and mailing address of the international preliminary examining authority: European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized Officer Huenges, A Telephone No. +49 89 2399-2280

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/EP 03/00546

I. Basis of the report

1. With regard to the **elements** of the international application *(Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17))*:

Description, Pages

1-4, 6-8, 10	as originally filed
5, 9	received on 11.03.2004 with letter of 08.03.2004

Claims, Numbers

1-15	received on 11.03.2004 with letter of 08.03.2004

Drawings, Sheets

1/4-4/4	as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

   These elements were available or furnished to this Authority in the following language: , which is:

   ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).

   ☐ the language of publication of the international application (under Rule 48.3(b)).

   ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

   ☐ contained in the international application in written form.

   ☐ filed together with the international application in computer readable form.

   ☐ furnished subsequently to this Authority in written form.

   ☐ furnished subsequently to this Authority in computer readable form.

   ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

   ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

   ☐ the description, pages:

   ☐ the claims, Nos.:

   ☐ the drawings, sheets:

5. ☒ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)).

   (Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

   see separate sheet

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	1-15
	No:	Claims	
Inventive step (IS)	Yes:	Claims	1-15
	No:	Claims	
Industrial applicability (IA)	Yes:	Claims	1-15
	No:	Claims	

2. Citations and explanations

   see separate sheet

Re Item I

Basis of the report

The amendments filed with the letter dated 08.03.2004 introduce subject-matter which extends beyond the content of the application as filed, contrary to Article 34(2)(b) PCT. The amendments concerned are the following:

- in claims 1-3 the expression "diffractometer" has been replaced by "equipment"; though the application as originally filed only refers to a diffractometer; there is therefore no basis for the generalization of the diffractometer to "equipment".
- dependent claim 7 refers to "any of the preceding claims" although only claim 1 refers to the rotation around the equatorial axis; therefore the reference to the other claims goes beyond the content of the application as originally filed.
- in claim 9, a pointing device is defined for positioning "the analytical unit" with respect to an element to be analysed. Yet, on page 7, pointing devices are described for positioning the **instrument** and not just the analytical unit with respect to the element under investigation. A pointing device for positioning only the analytical unit is not disclosed originally.

The opinion on novelty and inventive step (see Item V below) has been given as if the above-mentioned amendments had not been made.

Re Item V

Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Reference is made to the following document:

D1: GB-A-2 198 920 (UNIV MOSKOVSK) 22 June 1988 (1988-06-22)
D2: US-A-5 014 293 (BOYD DOUGLAS P ET AL) 7 May 1991 (1991-05-07)
D3: US-A-4 922 512 (LAJUS PIERRE ET AL) 1 May 1990 (1990-05-01)
D4: EP-A-1 016 375 (GEN ELECTRIC) 5 July 2000 (2000-07-05)
D5: US-A-6 064 717 (ORTEGA RICHARD ET AL) 16 May 2000 (2000-05-16)
D6: US-A-4 769 832 (LOUIDAY ANDRAE E) 6 September 1988 (1988-09-06)

2. Novelty and inventive step

2.1. Independent claims 1 and 12

The subject-matter of independent **claims 1 and 12** fulfills the requirements of the PCT with respect to novelty and inventive step for the following reasons.

Regarding claim 1, document **D1** discloses a diffractometer (see page 1, lines 3-13) comprising:
- an analytical unit (5, 6, fig. 1) supporting a radiation source (5, fig. 1), having a collimation axis (see fig. 4), and a radiation beam detector (6, fig. 1), having a reception axis (see fig. 4) converging in a centre (S, fig. 4) of the diffractometer which is fixed with respect to said analytical unit (see fig. 4);
- means for moving said analytical unit in the space (page 7, lines 9-18)
- means for rotating said source and detector around said centre of the diffractometer (page 7, lines 9-18), so that said collimation axis and said reception axis are kept in an equatorial plane, fixed with respect to said analytical unit (in D1 the □/2□ circle of fig. 4 represents the equatorial plane which goes through the position of the sample S and comprises all possible positions of the source and detector; this equatorial plane is kept fixed with respect to the analytical unit 5, 6, fig. 1)
- a support and movement structure (2, 3, fig. 1) supporting said analytical unit (5, 6, fig. 1)
- means (7, 8, fig. 1) for moving said analytical unit (5, 6, fig. 1) with respect to said support and movement structure (2, 3, fig. 1),
so that the analytical unit (5, 6, fig. 1) can rotate around an equatorial axis (in D1 the equatorial axis corresponds to the z-axis in fig. 4; when stage 2 is rotated around element 3, fig. 1, the analytical unit, 5, 6, fig. 1 is rotated around the equatorial axis being perpendicular to base 1), the equatorial axis being contained in said equatorial plane (see fig. 4: the z-axis is in □/2□ plane) and passing through the centre of the diffractometer (the z-axis in fig. 4 goes through the sample position S which is the center of the diffractometer).
The subject-matter of claim 1 differs therefrom in that the means for moving the analytical unit with respect to the support and movement structure permit the rotation of the "equatorial plane" (see the clarity objections under point 4 below) around the equatorial axis, without that the equatorial axis changes its position in

space.
The problem to be solved by the present invention may be regarded as the provision of a diffractometer with additional freedom of movement so as to examine a stationary sample in three dimensions.
None of the other documents cited in the search report discloses or hints at the provision of moving means permitting the rotation of the analytical unit - which defines the equatorial plane - around the equatorial axis, without that the latter changes its position in space.
Documents D2 and D3 show tomographic X-ray scanners comprising an X-ray source, a transmission detector assembly and a plurality of rotatable arms. However, there are no means provided for displacing the X-ray source with respect to the detector so as to measure diffraction of the beam.
Document D4 discloses a gantry with a source and a detector moveable thereon. Yet, like in documents D2 and D3, the detector is only arranged for detecting transmitted radiation.
Document D5 shows an X-ray diffractometer with an X-ray source and a detector which can be moved independently from one another by the use of two different robot arms which allow unrestricted motion in space. The source and detector are not placed on an arm such that their radiation axes meet in the center of the diffractometer (i.e. the sample position).
Document D6 discloses two cradles connected to each other for performing isocentric absorption measurements. Again there is not provided the possibility of performing diffraction measurements.

The subject-matter of claim 1 is therefore novel and inventive, Art. 33(2) and (3) PCT.

The subject-matter of claim 12 describes the use of the apparatus of claim 1 with the center of the diffractometer being positioned on a point of the surface of an element to be analysed. As the apparatus described in claim 1 is novel and inventive, so is the method of using this apparatus. Hence claim 12 is novel and inventive, Art. 33(2) and (3) PCT.

2.2. Dependent claims 2-11 and 12-15

Claims 2-11 and 12-15 are dependent on claims 1 and 12, respectively and therefore also fulfill the requirements of the PCT with respect to novelty and inventive step.

3. Industrial applicability

The industrial applicability of the subject-matter of **claims 1-15** is beyond doubt, Art. 33(4) PCT.

4. Clarity

The definition of the subject-matter of **claims 1 and 12** is unclear (Article 6 PCT). In the characterizing portion of claim 1 and in claim 12, means are defined permitting the rotation of the equatorial plane around the equatorial axis. However, the rotation of the equatorial plane does not clearly define which parts of the diffractometer are actually moved. The equatorial plane only represents a theoretical plane describing the position of the analytical unit and of the irradiation and detection axes. The rotation of the equatorial plane being a theoretical construction does not imply the rotation of any constructional part of the apparatus. For assessing novelty and inventive step the claims are interpreted such that the analytical unit is rotated around the equatorial axis.

that includes the analytical unit, supported by the arm (4). Locking devices (5) permit to fix the arm (4) positioned with respect to the support (3). The extremity (6), also visible in the Fig. 2 and Fig. 3, includes a x- ray source (7), a x-ray detector (8) and other positioning devices. These devices include the element (9), called primary Euler cradle, which may advantageously be in the form of a circular arch, devoted to support the x-ray source (7) and the detector (8). In the described case, the primary Euler cradle is the analytical unit. Source (7) and detector (8) can be conveniently moved along the primary Euler cradle (9). For each position reached on the primary Euler cradle by source and detector, the source collimation axis (10) and the reception axis (11) are always directed towards a point (12), which is the centre of the diffractometer (12) and can advantageously coincide with the centre curvature of the primary Euler cradle (9).

The axes (10) and (11), can thus rotate around the centre (12) in a plane, the equatorial plane, that is substantially parallel to the primary Euler cradle (9). In the Fig. 3 the equatorial plane coincides with the plane of the drawing, the axial plane is perpendicular to it, their intersection is the axis (13), called exploration axis.

According to a preferred embodiment of the invention, said primary Euler cradle (9) is conveniently supported by a structure supporting and movement (14), called secondary Euler cradle. A special system permits to the primary Euler cradle (9) to be moved with respect to the secondary Euler cradle (14) to execute a rotation around the equatorial axis (15). This equatorial axis (15) is contained in the equatorial plane and is perpendicular to the exploration axis (13). In this way, the whole equatorial plane can rotate of a certain angle with respect to the equatorial axis (15), and thus the collimation axis (10) and the reception axis (11) can rotate because the source (7) and the detector (8) are supported by the primary the Euler cradle (9).

Fig. 4 shows a lateral view of the extremity (6) that includes the two Euler cradles, and shows a possible implementation of the articulation mechanism of the primary Euler cradle (9) with respect to the secondary Euler cradle (14). The primary Euler cradle (9) includes two cog arcs (21) and (21'), suitably joined. The source (7) and

called specimen plane, shall be perpendicular to the exploration axis. Thus the collimation axis (10) forms an angle θ with the specimen plane. The reception axis (11) will form an angle θ with the specimen plane and 2θ with respect to the collimation axis. The system is thus able to detect the rays reflected by families of crystallografic planes, that have a interplanar distance d that , for an angle θ correspondent to the relative position of the source and detector, satisfies the Bragg's law $n\lambda=2d*\sin\theta$, where n is a whole number and • the wavelength of the x- ray beam emitted from the source.

According to a possible operating method, the collimation axis (10) and the reception axis (11), perform the above mentioned rotation by keeping themselves symmetric with respect to the exploration axis (13); thus, it is possible to detect the diffraction beam from various families of lattice planes satisfying the Bragg law at different angles θ.

When the specimen is a polycrystalline solid with enough small crystals, as it is common, the various families of planes may be randomly oriented in all the directions. So by scanning various angles θ, the various families of planes that satisfy the Bragg law can be detected. By a rotation of the equatorial plane, around the equatorial axis (15), as above mentioned, and by keeping unvaried the position of the source and detector with respect the axis of exploration (13) (that will be rotated of ω together with the equatorial plane), The equatorial plane will be no longer perpendicular to the specimen plane. It is thus possible to scan again the different angles θ, and detect signals from the planes inclined of an angle ω with respect to the specimen plane. The comparison at different θ angles of diffraction intensities at the same θ angle (corresponding to plane families with the same interplanar distance), give an information on the possible preferred orientations in the crystalline structure. This is equivalent to explore for a certain arc the Debye circle.

Alternatively, the collimation and reception axes can be kept symmetrical with respect to an axis laying on the equatorial plane and different from the exploration

CLAIMS

1. Diffractometer comprising:

- an analytical unit (9) supporting a source (7) of a radiation beam, having a collimation axis (10); and a radiation beam detector (8) having a reception axis (11), said collimation (10) and reception (11) axes converging in a centre of the diffractometer (12), which is fixed with respect to said analytical unit (9);
- means (16, 31, 32, 33) for moving said analytical unit in the space;
- means (20, 20') for rotating said source and detector around said centre of the diffractometer.

2. Diffractometer according to claim 1, wherein the source (7) is a x-ray source and the detector (8) is a x-ray detector.

3. Diffractometer according to claim 1 or 2, wherein said means (16, 31, 32, 33) for moving said analytical unit (9) in the space, are suitable to permit to change the position of said centre of the diffractometer (12).

4. Diffractometer according to any of the preceding claims, wherein said means (20, 20') for rotating said source (7) and detector (8) are suitable to rotate source and detector, so that said collimation axis (10) and said reception axis (11) are kept in an equatorial plane, fixed with respect to said first analytical unit (9).

5. Diffractometer according to any of the preceding claims, wherein said analytical unit (9) is supported by a support and movement structure (14) and means (27) are provided for moving said analytical unit with respect to said support and movement structure (14), so that the analytical unit (9) can rotate around an equatorial axis (15) contained in said equatorial plane and passing through said centre of the diffractometer (12).

6. Diffractometer according to claim 5, wherein said equatorial axis (15) is perpendicular to a symmetry plane of said analytical unit (9).

7. Diffractometer according to claim 5 or 6, wherein said means for moving said analytical unit (9) with respect to said support and movement structure (14), permit the rotation of the equatorial plane with respect to said equatorial axis (15), without that the latter changes its position in the space.

8. Diffractometer according to claim 7, wherein said rotation is possible along an arc of at least 10°, preferably at least 20°.
9. Diffractometer according to claim 2, wherein said detector (8) is a proportional ionisation counter.
10. Diffractometry method comprising positioning a diffractometer comprising:
- an analytical unit supporting a source of a radiation beam, having a collimation axis and a radiation beam detector having a reception axis, said collimation and reception axes converging in a centre of the diffractometer, which is fixed with respect to said analytical unit ;
- means for moving said analytical unit in the space;

means for rotating said source and detector around said centre of the diffractometer;

with said centre of the diffractometer on a point of the surface of an element to be analysed.
11. Method according to claim 10, wherein said analytical unit has a symmetry plane and said plane is placed perpendicularly to the surface of the element to be analysed at the point coincident with said centre of the diffractometer.
12. X-ray diffractometry method according to claim 10 or 11.
13. Method according to any claim from 10 to 12, wherein said element to be analysed is not mechanically linked to the diffractometer.